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SOME STUDIES ON SOLUTIONS OF CARO'S ACID AND POTASSIUM PERSULPHATE

by

WILLARD WELLINGTON HODGE

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INTRODUCTION.

In 1834 Faraday[1] stated that during the electrolysis of concentrated solutions of sulphuric acid there was a large loss of oxygen unaccounted for. This loss occurred around the anode. He thought it might be due to the formation of hydrogen peroxide, but never investigated the matter further, and the question remained unsettled until 1878.

In that year Berthelot[2] prepared persulphuric acid. He first prepared persulphuric anhydride by passing silent discharges thru an ozone tube filled with a mixture of sulphuric anhydride and oxygen. The white crystalline substance formed in the tube he called persulphuric anhydride S_2O_7. To the aqueous solution of this substance he gave the name persulphuric acid and assigned the formula $H_2S_2O_8$. At about the same time, he noted the formation of a similar substance around the anode during electrolysis of concentrated (40% or more) sulphuric acid. This partially explained the phenomena first noticed by Faraday nearly a half century earlier and began the study of persulphuric acids.

Moritz Traube[3] later studied the action around the anode plate in the electrolysis of concentrated solutions of sulphuric acid. From his results he concluded

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1. Trans. Philos. (1834) p. 91.
2. Ann.Chim.Phys. (1878)(5)14,345; (1880)(5)21,176,193,286.
2. Comptes Rendu, (1878)86,71,277; (1880)90,269,331.
3. Ber. (1889)22,1518; (1891)24,1764; (1892)25,95; (1893)26,1481

that the substance formed was not an acid or an anhydride but a tetraoxide of sulphur SO_4. He named this substance the holoxide of sulphur.

The difference in opinion of these two investigators led to considerable discussion of the subject in the literature current at that time. Mendeleeff[1.] favored the views of Traube. He supposed that the substance was a peroxide of sulphur in many ways similar to barium peroxide, hydrogen peroxide, etc. and that it would not form salts.

The discussion was ended and the explanations of Traube and Mendeleeff disproved when Marshall[2] in 1891 prepared a series of well defined salts, by the electrolysis of concentrated solutions of the corresponding acid sulphates. Analysis of these salts gave them the general formula $R_2S_2O_8$.[3.] tho for the potassium salt Walker[4.] obtained the molecular formula KSO_4.

Little further work on the higher oxygen acids of sulphur is recorded for some time. Armstrong[5.] in his pressidential address(1895) referred to the unsatisfactory

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1. Bull. Soc. Chim. 38, 168.
2. Journ. Chem. Soc.(1891) 771
2. Proc. Royal Acad. Edin. 18, 63.
3. Marshall,
   . Berthellot, Comptes Rendu,(1891)112,1481.
   Ann. Chim.Phys.(1892)(6)26,526.
4. Proc. Royal Soc. Edin. (1891)18,64.
5. Pres. Address, (1895) 6,1156.

condition of our knowledge concerning the persulphuric acids. a condition which still exists.

In 1898, Caro[1] was studying the action of persulphates on aniline. He observed oxidizing effects that he could not account for from the present knowledge of the reagents used.

The phenomena noticed by Caro was the incentive to further investigations on the higher acids of sulphur. Baeyer and Villiger[2] prepared solutions of potassium persulphate with sulphuric acid, and hydrogen peroxide with sulphuric acids and gave the name, Caro's Acids to these reagents. They used them to study the oxidizing effect on several organic compounds. As a result they secured the superoxides of several of the substances and came to the conclusion that the formula for Caro's acid was either H_2SO_5 or $H_2S_2O_9$. A number of investigators working along lines similar to those above referred to, came to about the same conclusions. Lowery and West[3] worked out the equilibrium existing in a system of sulphuric acid, water, persulphuric acid and hydrogen peroxide. They tried different concentrations and recorded observations at different temperatures. They concluded that a series of sulphur acids existed the general formula being $H_2O_2.xSO_3$. To the

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1. Caro Zeit. Angew. Chem. (1898),845.
2. Ber. (1899)32,625;(1900)33,124,858,1569,2479,2488; (1901)34,853.
3. Journ. Chem. Soc. (1900),950.

highest of these acids they gave the formula $H_2S_4O_{14}$ and others might be $H_2S_2O_8$, $H_2S_2O_7$. The series could be represented by the equation,

$$\frac{C^1}{C^2} = K_1\left(\frac{C_3}{C_4}\right)^2 + K_2\left(\frac{C_2}{C_4}\right)^4.$$

They rejected the formula H_2SO_5 for the curve plotted could not correspond to any such acid. Bach[1] favored the formula H_2SO_5. Armstrong and Lowery[2] secured some results indicating a series of acids, H_2SO_4, $H_2S_2O_6$, $H_2S_2O_8$, and $H_2S_4O_{14}$, and perhaps a peranhydro acid(Caro's) H_2SO_5.

The work of T.S.Price[3] is of especial interest. He obtained results showing that the acid was either H_2SO_5, monobasic, or $H_2S_2O_9$, dibasic and could not be $H_2S_4O_{14}$. In his latest work he tried to prepare the potassium salt of Caro's acid by electrolysis. He obtained a mixture of potassium persulphate, hydrogen peroxide, potassium acid sulphate and a residue which on analysis favored the formula H_2SO_5 for Caro's acid. He worked with dilute solutions at low temperatures.

Ahrle[4] in 1909, synthesized Caro's acid from 100 per cent hydrogen peroxide and sulphuric anhydride. From

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1. Ber. (1900)33,3111.
2. Proc. Roy. Soc. (1902)70,94.
3. Journ. Chem. Soc. (1903)83,543; (1906)89,53;(1905)21,299
   Ber. (1902)35,292.
   Price & Friend, Journ. Chem. Soc.(1904)85,1526.
   Price & Deming, Zeit. Phys. Chem. (1903)46,101.
4. Journ. f. Pk. Chem. (1909)79,129.

the reactions of this substance, he assigned the formula H_2SO_5 to Caro's reagent.

In looking over the material on the persulphuric acids we note that Price stated the theoretical basis for the work most clearly. Many of the authors referred to, have adopted nearly similar working bases. Some of the work has been done with dilute solutions, some with concentrated. High and low temperatures have been employed. The oxygen liberated has been determined with potassium permanganate and by the iodimetric method. Discrepencies also are found because the action of sodium hydroxide and barium hydroxide do not give the same results when the acidity is determined. We thought some work with the aqueous solution of the compounds used in making Caro's acid might give some light on the subject so performed the following experiments.

THEORETICAL CONSIDERATIONS[1].

The difference between an acid of the formula H_2SO_5, $H_2S_2O_9$ and $H_2S_4O_{14}$ would be recognized from the ratio of the substances formed in the reactions producing the acid.

1. If the formula is H_2SO_5 we have

$$K_2S_2O_8 + H_2SO_4 = H_2S_2O_8 + K_2SO_4$$

On addition of water this would give

$$H_2S_2O_8 + H_2O = H_2SO_5 + H_2SO_4.$$

2. If the formula is $H_2S_2O_9$ we have

$$2K_2S_2O_8 + H_2SO_4 = 2H_2S_2O_8 + 2K_2SO_4.$$

adding water

$$2H_2S_2O_8 + H_2O = H_2S_2O_9 + 2H_2SO_4.$$

3. If the composition is $H_2S_4O_{14}$ we have the reaction

$$2K_2S_2O_8 + 2H_2SO_4 = 2H_2S_2O_8 + 2K_2SO_4.$$

the persulphuric acid breaking up into Caro's acid and hydrogen peroxide.

$$2H_2S_2O_8 = H_2S_4O_{14} + H_2O_2.$$

If the reaction takes place according to equation 1, the ratio of iodine value to increase in acidity would be 254:98=2.59;

If according to equations 2, we have the ratio 508:98=5.18;

If as represented in equations 3, the ratio between iodine liberated and hydrogen peroxide found is 254 : 34 = 7.47

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[1] Ber.(1902)35,291; Jr. Ch. Soc.(1903)83,543 Bay.& Villiger

EXPERIMENTAL PART.

In the experimental part of the work we observed all the usual precautions to ensure accuracy. The burettes and pipettes were calibrated and the weights used, checked. Phenolphthalein was used as an indicator in determining the acidity and special precaution was taken to free the water used, of all carbonate.

The following solutions were then prepared.

Solution No. 1. This was a solution of Caro's acid prepared as by ~~the~~ Baeyer and Villiger, Price and others. (loc. cit.) Four grams of potassium persulphate were dissolved in 10c.c. of concentrated sulphuric acid at the temperature of a mixture of ice and salt. This was made up to 500c.c. by slowly adding ice water.

Solution II. Four grams of potassium persulphate were made up to 500 c.c. with ice water..

Solution No. III. Ten c.c. of concentrated sulphuric acid was made up to 500c.c. again using cold redistilled water.

Thus solution II gives us practically the same concentration of potassium persulphate as solution No.I. Also solution III should give us the same acidity as solution I unless another acid is formed in solution I.

One way of distinguishing Caro's acid from persulphuric acid is the difference in the rapidity with which they liberate iodine from potassium iodide solution. To obtain this difference in velocity we performed

a series of time titrations with the results given in table A.

In this experiment 20 c.c. of N/10 potassium iodide solution was placed in each tube. To each tube 5 c.c. of one of the reagents was added. The solutions were measured out at 15°C. They were allowed to stand for the length of time indicated. The iodine liberated was determined and then the acidity. Iodine liberated is given in grams iodine. Acidity is expressed in grams sulphuric acid.

TABLE A.	Solution I.		Solution II.	
Time	I liberated	Acidity	I liberated	Acidity
30 sec.	0.02718 gms.	0.31894 gms.	0.00066 gms.	0.00226 g.
60 "	0.02751 "	0.32168 "	0.00252 "	0.00226 "
5 min.	0.03069 "	0.31555 "	0.00368 "	0.00226 "
10 "	0.03540 "	0.31894 "	0.00640 "	0.00241 "
15 "	0.03646 "	0.31924 "	0.00788 "	0.00271 "
30 "	0.03534 "	0.31796 "	0.01843 "	0.00255 "
1 hrs.	0.03686 "	0.31947 "	0.02435 "	0.00277 "
2 "	0.03792 "	0.31872 "	0.02957 "	0.00241 "
3 "	0.03766 "	0.31947 "	0.03368 "	0.00249 "
4 "	0.03753 "	0.31939 "	0.03440 "	0.00309 "
5 "	0.03769 "	0.31826 "	0.03564 "	0.00264 "

Solution III liberated no iodine and had a uniform acidity of 0.30631 grams.

From the table we see that solution I liberated slightly more iodine than solution II. We therefore tested the solutions with titanium sulphate. Solution I showed a trace of hydrogen peroxide, the other solutions showed none present. This accounts for the excess of iodine liberated by the Caro's acid.

To explain the results obtained in this experiment we might make two assumptions. First: that in solution I the Caro's acid present immediately liberates a large amount of iodine and then the persulphuric acid present slowly liberates more iodine until a state of equilibrium is reached. This took about[1] two hours at the room temperature, 24°C. Or secondly, we might assume that the sulphuric acid or hydrogen peroxide present in solution I. acted catalytically(Loewenhart[1]) thus liberating more rapidly the iodine which an aqueous solution of potassium persulphate would free if given a longer time in which to act. In this case about four hours whereas solution I reached the same state of equilibrium in two hours.

To test the validity of our second assumption we performed the following experiment. It is a well known fact that an increase in temperature usually accelerates chemical action. Therefore if at a higher temperature solution II will reach the state of equilibrium in the same time as solution I, we might say that some reagent in

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[1] Amer. Chem. Journ. (1903)29,563.

solution I. acted catalytically. If we found that solution II. became acid we might assume that the catalytic action was due to this acid. But if it did not we would have to assume that the increased velocity of reaction was due to the heat alone changing the relations of the ions in the solution.

As in the previous experiment 20c.c. of N/10 potassium iodide was placed in each of several test tubes. Five c.c. of solution II. was added to each. The tubes were placed in a beaker of boiling water. The temperature fell slightly on account of adding the tubes, but boiled again in a moment. At the end of the time indicated the tubes were removed and contents titrated as for table A.

TABLE B. Solution II.

Time.	Iodine liberated.	Acidity after I liberated.
......	....	0.00166 gms.
2 min.	0.02944 gms.	0.00151 "
3 "	0.03143 "	0.00136 "
5 "	0.03372 "	0.00128 "
7 "	0.03385 "	0.00098 "
10 "	0.03567 "	0.00090 "
12 "	0.03534 "	0.00090 "
15 "	0.03440 "	0.00113 "

From these results we see that at a higher temperature solution II will liberate just as much iodine from a potassium iodide solution as Caro's acid and that

it will do it more quickly . Here a state of equilibrium is reached in twelve minutes while with solution I. the action continued for two hours. Furthermore there is no increase in acidity so we cannot say that the iodine is liberated by a higher oxygen acid of sulphur unless this acid is decomposed by the liberation of the iodine. From the above table we could look upon the reaction according to the simple equation;

$$KSO_4 + KI = K_2SO_4 + I$$

It might be thought iodine would be evolved from the potassium iodide solution alone at the higher temperature. We tried the experiment obtaining negative results.

From these results it would appear that neither persulphuric nor Caro's acids are necessary to liberate iodine from potassium iodide solution if potassium persulphate is present. This would seem to prove our second assumption, viz., that the sulphuric acid in solution I. acts merely as a catalytic agent serving the same purpose as the higher temperature used in experiment B. If, however, a solution of potassium persulphate without potassium iodide present increases in acidity subjected to the higher temperature of Experiment B. then we could look upon the iodine liberated as being freed by the decomposition of the acid. The acid being decomposed as rapidly as formed when potassium iodide is present during its formation.

Or, secondly, if we obtain a difference in acidity of solution I.before and after the iodine isliberated .we can look upon the iodine as being liberated by an acid formed in the reaction.

To check these two explanations, we made up some fresh solutions I, II, and III. As the temperature rose, bubbles of gas were noticed coming off from solution I. We decided to allow the reaction to be completed before using the solutions. So the solutions were placed in a refrigerator and kept at a temperature of 12° to 15° for two weeks. No more gas was now evolved from solution I. These solutions were tested for changes of acidity also for any new end products to the reactions.

First, 5 c.c. of solution II. were placed in each of several test tubes. The tubes were placed in boiling water and the contents tested at the periods indicated.

TABLE C. Solution II.

Time	Acidity	Time	Acidity
.....	0.00173 gms.	15 min.	0.01206 gms.
2 min.	0.00204 "	30 "	0.01696 "
3 min.	0.00369 "	60 "	0.02224 "
5 "	0.00462 "	90 "	0.02688 "
7 "	0.00550 "	2 hrs.	0.02601 "
10 "	0.00639 "	3 "	0.02688 "

The results show an increase in acidity. In table B we obtained no increase in acidity when potassium

iodide was present, but the liberation of considerable iodine. The acidity here increased for an hour and a half which is about the same length of time as solution I. table A took to reach a state of equilibrium. In table B the total iodine value was attained in twelve minutes. In other werds, if the liberation of iodine in experiment B was due to the decomposition of the acid formed as in experiment C the reaction was completed in about one sixth the time when the potassium iodide was present in the solution of potassium persulphate. Now if the liberation of iodine shown in table B was due to the acid formed as observed in table C then when the acid is formed on adding the potassium iodide we should expect the iodine to be immediately set free. We tried it with duplicate tubes of the ninty minutes and two hour periods but no iodine whatever was liberated. We may look upon the acid formed in experiment C as sulphuric acid and the reaction as

$$2KSO_4 + H_2O = K_2SO_4 + H_2SO_4 + O$$

The oxygen liberated iodine in experiment B when the potassium iodide was present during the reaction, but was driven off by the heat in experiment C so was not present in the solution when the acidity reached its maximum. This would explain why the iodine is liberated in the first case and not in the second, where the potassium iodide solution is not added until the acid formation is completed. From the above experiments we must conclude that the sub-

stances formed and the velocity of their reaction varies with the temperature of, and the solutes present in the solutions.

To further investigate the second explanation offered above we performed the following experiment. The solutions were used the same as for table A only they were allowed to stand over night.

TABLE D. Solution I.

	Acid before	I liberated	Acid after
	0.32457 gms.	0.04177 gms.	0.30778 gms.
	0.32627 "	0.04110 "	0.30692 "
	0.32580 "	0.04124 "	0.30816 "
	0.32626 "	0.04192 "	0.30839 "
Mean	0.32595 "	0.04150 "	0.30763 "

Solution II.

Acid before	I liberated	Acid after
0.00136 gms.	0.03673 gms.	0.00181 gms.
0.00143 "	0.03626 "	0.00173 "
0.00143 "	0.03646 "	0.00181 "
0.00129 "	0.03666 "	0.00188 "

Three facts in this table deserve attention. First, there is more iodine liberated by solution I. than when it was first made up. Secondly, there is a decrease in acidity on liberation of the iodine. Thirdly, solution II, has about the same value as when first prepared. The slight

increase in acidity of solution II. could be accountedfor by absorbing carbon dioxide from the air on standing overnight. Phenolphthalein was used as an indicator.

To account for the first fact, the titanium sulphate test showed considerable hydrogen peroxide present. The mean of several determinations with potassium permanganate gave the oxygen in solution I. due to hydrogen peroxide an iodine equivalent of 0.00809 gms..This subtracted from the mean value 0.04150 gms. given in table D leaves 0.03341 gms. as the iodine liberated by the acid present. This is somewhat lower than results given in table A.

To make final checks on the completeness of the reactions solutions were prepared as in the previous experiment and allowed to stand in the refrigerated for forty hours. The mean of four determinations with each solution is given in table E.

TABLE E.

	Acid. before	I liberated	Acid. after
Solution I.	0.33761 gms.	0.03792 gms.	0.30620 gms.
Solution II.	0.01749 "	0.03487 "	0.05402 "
Solution III.	0.30612 "	0.00000 "	0.30632 "

Solution I. H_2O_2 = 0.00681 gms.

From this series of investigations we can state the following conclusions, subject of course to the conditions under which these experiments were performed.

First that equivalent solutions of potassium per-

sulphate have about the same oxidizing value as Caro's acid. But that the persulphate solutions require about twice as long a time at 22°C. to reach a state of equilibrium.

Secondly, that the immediate liberation of a large per cent of iodine by Caro's acid could be looked upon as a catalytic action due to the presence of hydrogen peroxide[1] or sulphuric acid. For the same results can be obtained by raising the temperature of the potassium persulphate solution which contains neither hydrogen peroxide nor sulphuric acid.

Thirdly, the liberation of the iodine may be due to a partial reduction of an acid of the formula H_2SO_5. since there is an increase in acidity in making Caro's acid. The ratio between this increase in acidity and the iodine liberated is, Table E. 0.03141:0.03111::1:1 approximately. That is for each molecule of the new acid formed there is a molecule of iodine liberated. Also Caro's acid after liberating the iodine has the same acidity as a solution made up with sulphuric acid and water in the same proportions. We may suppose the additional molecule of oxygen loosely linked to the molecule of sulphuric acid present as $H_2SO_4 = O$ or $\begin{matrix}H_2SO_4\\H_2SO_4\end{matrix}\!>O$ That is having certain aggregate[2] molecules in the solution. We could still look

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[1] Loewenhart, Amer. Chem. Journ. 29, p. 563
[2] Moore, Journ. Chem. Soc. 93, p. 2179-

upon the sulphuric acid as a catalytic carrier or temporary retainer of the additional oxygen molecule as the solution is quite unstable. For even at 15°C. it slowly decomposes forming some hydrogen peroxide and a more stable acid of sulphur.

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